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Carbon Fiber Counting

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CARBON FIBER COUNTING

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SUMMARY

A method has been developed for characterizing the number and lengths of carbon fibers accidentally released by the burning of composite portions of civil aircraft structure in a jet fuel fire after an accident. The method was developed as part of a Langley Research Center assessment of the risk associated with such release. Representative samplings of carbon fibers collected on transparent sticky film were counted from photographic enlargements with a computer-aided technique which also provided fiber lengths. Comparisons have been made between the Langley method and the methods used by three other laboratories through a round-robin counting of 12 selected sticky sampler records. Consistent results were obtained among counts from Langley and two of the other laboratories. A major source of counting error was found in the technique of the fourth laboratory which, when corrected, placed their fiber counts within the same range as the others.

INTRODUCTION

The Graphite Fibers Risk Analysis Program Office at Langley Research Center has been charged with assessing the risk associated with the accidental release of carbon fiber which might occur from the burning of composite portions of a civil aircraft structure in a jet fuel fire during or after an accident. Determining the number and size of carbon fibers released from a burned composite was a major part of the investigation that was necessary in order to determine the risk of electrical or electronic equipment malfunctions or damage from short circuiting if airborne carbon fibers settle on electrical contacts.

Composite plates and structural elements were burned in laboratory experiments under a variety of conditions which influence the release of single fibers. Generally, representative portions of these fibers were collected on sticky samplers which then were inspected to determine quantity and size. Similar samplers have been used with the few outdoor experiments conducted. Because of the generally short lengths and small diameters of single carbon fibers released from burned structural composites, examination of collection samplers was done with visual magnification ranging from about 8 to 20X. The number of fibers deposited on any given sampler ranged from zero

N80-28446 #

up to tens of thousands, depending on the particular conditions of the composite burn and the location and size of the sampler. Counting large numbers of fibers is tedious and becomes more difficult when large quantities of multiple fiber clumps and composite laminae strips are intermingled with the single fibers. Soot particles released from incomplete combustion associated with large pool fires of jet fuel increase the difficulty of distinguishing single fibers.

Several techniques were therefore developed for efficient counting of the single fibers by various organizations, based on their experience and on the type of records available for counting. Generally, these techniques included some form of sampling from the total record, since it is impractical to count the large numbers of fibers on the entire sample. However, large variations were observed in the measured results and it was suspected that some of the variations might be due to differences in counting techniques rather than to differences in fiber release conditions. Therefore, a round-robin counting of selected sticky paper records has been conducted under the auspices of the Naval Surface Weapons Center, Dahlgren, Virginia. Four laboratories which were actively involved in counting fibers in the joint DOD-NASA carbon fiber risk analysis program participated in the round-robin.

This report describes the method used to count and to characterize carbon fibers at the Langley Research Center and presents the results from the counting of the selected round-robin samples. Overall results of the risk assessment were presented at an industry/government briefing held December 4-5, 1979, at the Langley Research Center (ref. 1).

FIBER COUNTING METHODOLOGY

Small sticky cylinders were used to measure carbon fiber exposure levels for many of the experiments conducted at the Langley Research Center on the vulnerability of electrical equipment (ref. 2). These cylinders were constructed from 50 mm squares of 0.5mm thick adhesive-coated polyester film. The transparent film was rolled, adhesive side out, on a 13mm diameter mandrel to form the cylinders. The overlapping edges of the film were joined by the adhesive coating. The cylinders were supported in a flow stream by attaching the overlap region to a wire strut. After exposure, the cylinders were cut at the overlapping joint, unrolled, and pressed, adhesive-side-down, against a microfilm aperture card (a computer card with a transparent film-covered opening 35 mm x 48 mm).

Fibers were counted by direct examination of a 35mm x 35mm square area of the mounted sticky film through a stereo microscope at 20X magnification. In the microscope with oblique

front illumination, carbon fibers were easily distinguished from other forms of fibrous contamination and from scratches on the transparent sticky material. For light deposits of fibers up to about 50 fibers on a 35-millimeter square (4×10^4 fibers/m²) counting the total area of the sampler was done easily with the microscope. However, for heavier deposits of fibers, or if fiber lengths were to be determined, an alternate method was developed.

An enlarged photographic print was made from the aperture-card-mounted sticky sampler with the same microfilm printer that is used to reproduce full-size copies of engineering drawings after they have been microfilmed. The 20X enlargement was then placed on a magnetic digitizer board and the fibers were identified to a computer by touching the ends of their images with a magnetic pen. A computer program computed the lengths of fibers from the coordinates of the identified fiber ends, and at the completion of scanning the photograph, the computer printed a listing of all the fibers in a length-ordered sequence from shortest to longest.

Single-fiber lengths down to about 0.1 mm can be clearly resolved; however, for the current investigation, only those fibers of lengths greater than one millimeter were considered since these were the only ones of interest as potential hazards in the short circuiting of electrical equipment.

ROUND-ROBIN COUNTING

The round-robin counting was planned with four selected sticky paper samplers from each of three tests conducted inside a closed chamber at the Naval Surface Weapons Center (NSWC), Dahlgren, Virginia (ref. 3). Graphite-epoxy plates were burned and an explosive charge was then detonated beneath the residue, yielding deposits of single fibers, multiple-fiber clumps, strips, and pieces. Three of these tests were selected for the round-robin counting as being representative of heavy, medium, and light deposition. The sticky-paper samplers in all these tests were flat transparent sheets of adhesive-coated polyester film 15 by 23 cm laid on the floor of the test chamber in a regular pattern in order to sample the deposition of fibers released in the test. Fiber clumps and strips are readily visible against a mottled background of soot and fine particles in a photograph of a typical NSWC chamber sticky sampler (fig. 1). Single fibers are essentially invisible at the magnification in this photograph but are more or less uniformly distributed over the entire surface.

In order to count these samplers by the Langley method, a 35 mm square piece was cut from the lower right corner of each sampler, (fig. 2) and mounted on an aperture card which was

then printed as a photographic enlargement at 20X. The photographs of the lighter depositions were counted over the entire area, whereas for the medium and heavy depositions, only about three percent of the photographed area was counted. In the photographs of the medium and heavy depositions, an area 12.7 cm (5 inches) square was marked near the center of the photograph (where fiber image contrast was the greatest) and the fibers in this area were counted. The count included fibers which were partially in and partially out of the square if they crossed either the left hand or the bottom boundary of the marked area.

As a check on the representative nature of the area counted, four adjoining areas were marked and counted on one of the medium deposition photographs and two adjoining areas on one of the heavy deposition photographs. The location of these areas is shown in figure 2.

RESULTS AND DISCUSSION

The results of the counting in the selected area of each of the photographed samplers are given in Table I. In each of the samplers, the actual area counted contained from 21 to 78 fibers with lengths greater than 1 mm. The projected total fibers on the 15 x 23 cm sticky were obtained by multiplying the actual fibers counted by the ratio of the total sticky area to the area counted. For the heavy and the medium depositions this multiplier was generally 864, while for the light depositions it was only about 30.

For the heavy deposit sample (number 20013, Table II), the two adjoining areas counted yielded projected total fiber counts of 31100 and 28500, which were within plus or minus 5 percent of the average of the two areas. For the medium deposit sample (number 20812), the four adjoining areas counted yielded projected total fiber counts ranging from 13800 to 19900 which were within plus or minus 18 percent of the four-area average of 16,800.

For the lighter deposition samples as much of the 35 mm square piece as could be photographed was counted. A quick scan of the entire sampler indicated apparently uniform deposition; however, because the deposition was so light, it was difficult to be certain of uniformity. A check was made on sample number 13884 by cutting a second 35 mm square from the lower left corner and counting its fibers. As shown in Table II, there was a 3 to 1 difference in the total count of fibers on the two areas taken as representative of this sampler, which can also be expressed as plus or minus 50 percent of the average. But the count on both of them compared with either medium or heavy deposits was low.

In addition to the total number of fibers counted, the distribution of the number of fibers by length increment was also determined (see Table III). The percentage of the total in each one-millimeter increment from 1 to 7 mm was tabulated for the individual samplers as well as for the average within each deposition category. Figure 3 presents these average distributions by length in graphical form. Although the longest fibers counted were in the 6-7 mm length range, the distribution was concentrated primarily in the 1 and 2 mm lengths, with a tendency to be more sharply peaked for the lighter deposition.

The mean lengths and the standard deviation of the fiber lengths are also given in Table III for each of the samplers counted. The consistency in these values is an indication of similarity within the distributions of fiber lengths for the various samplers selected for this round robin counting. Mean fiber lengths for fibers greater than 1 mm in length ranged from a high of 2.4 mm for a heavy deposition to a low of 1.5mm for a light deposition. This variation in mean fiber length with fiber release density may have been the result of the same differences in burn time, burn temperature, or carbon-fiber composite specimen construction that produced the variations in fiber release density. However, it was not a strong trend and these three tests were not sufficient in themselves to draw a conclusion concerning factors which may influence variations in average lengths of fire-released fibers.

Participants from the four laboratories involved in the round robin fiber counting met at the Langley Research Center May 10, 1979, to discuss the techniques used by each laboratory and to present data which had previously been submitted to the Naval Surface Weapons Center. (ref. 4). The four participating laboratories were Defense and Space Systems Group of TRW, Inc., Scientific Service, Inc., U. S. Army Dugway Proving Ground, and NASA-Langley Research Center. A summary of the fiber counting is listed in Table IV and is shown graphically in Figure 4 for the light, medium and heavy deposits.

All four laboratories obtained reasonably consistent total fiber counts for the samplers from the light deposition test (687 fibers average per sampler or 0.2×10^5 fibers/m²). All four used similar methods of total fiber counting within specified subdivisions of the total sampler area. The size of the area and method of location varied among laboratories.

For the medium (6.5×10^5 fibers/m²) and heavy (11.4×10^5 fibers/m²) deposition tests, three of the laboratories continued to use the total fiber counting methods. Dugway Proving Ground changed to a technique for counting based on an application of the Buffon needle drop problem (ref. 5). This technique provided an expedient and systematic method for counting when the fiber

density on the samplers was so great that overlapping and crisscrossing of fibers made total fiber counting difficult. However, in the round robin counting, this technique gave results which were consistently greater than the other three laboratories' total fiber counting methods. As can be seen, (fig. 4), the Dugway results were 2.8 and 2.3 times greater than the average of the other laboratory fiber counts for heavy and medium deposits.

Subsequent investigation at Dugway found that the grid lines printed on a transparent overlay used in the Buffon counting technique were too thick and biased the count, and in addition the people counting fibers were having difficulty determining which fibers to accept and which to reject when the length was close to the 1 mm limit. With length spectra similar to those in figure 3, there were many fibers with lengths close to 1 mm. The addition of 1 mm reference marks on the transparent overlay has helped resolve the uncertainty and Dugway has reported that a recount of the round-robin samplers with the reference marks and with thin grid lines has reduced their total fiber counts to a level consistent with the averages of the other three laboratories.

Scientific Service, Inc. (SSI) indicated that they could not analyze accurately the heavy deposit samplers with their total fiber counting method without first redistributing the fibers, thereby destroying the original samples.

CONCLUDING REMARKS

A method for counting and sizing by length single carbon fibers released from burning composite materials was developed at the Langley Research Center. This method used photographic enlargement and a computer-aided counting technique for characterizing 35 mm sticky collector samplers. The method has been used in a round-robin counting exercise among four laboratories on four selected sticky paper samplers from each of three tests representative of heavy, medium, and light deposits of carbon fibers. For adjoining areas on the same sampler, counting variations of about 5 percent from average were established for heavy deposits, 18 percent for medium deposits and 50 percent for the small quantity of fibers on the light deposit. The average fiber length for fibers greater than 1 mm in length ranged from 2.4 to 1.5 mm over all 12 samplers.

Round robin counting of single carbon fibers established reasonably consistent total fiber counts for light deposition samplers, 0.2×10^5 fibers/m². For medium (6.5×10^5 fibers/m²) and heavy (11.4×10^5 fibers/m²) deposition samplers, the Langley counting method gave total fiber counts that were consistent with two other laboratories using similar approaches. However, the expedient counting method adopted by Dugway Proving Ground

gave fiber counts that were two to three times greater than the average of the other laboratories. The Dugway method has been subsequently modified to better differentiate which of the fibers should be counted. With this modification the Dugway total fiber counts were reported to be consistent with the counts of the other laboratories.

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TABLE I.-SINGLE CARBON FIBERS COUNTED, AND PROJECTED TOTAL CARBON FIBERS ON STICKY SAMPLERS SELECTED FOR ROUND ROBIN COUNTING FROM THREE NSWC CHAMBER BURN AND EXPLODE TESTS

Test & Sampler number	Area counted	Fibers counted (a)	Projected total fibers on 15 x 23 cm sticky sampler
BT-230 Heavy deposition			
19968	40.3 mm ²	53	45800
19970	↓	47	40600
19980	↓	37	32000
20013	80.6	69	29800
BT-237 Medium deposition			
20803	40.3 mm ²	32	27600
20805	↓	23	19900
20810	↓	23	19900
20812	161.2	78	16800
BT-171 Light deposition			
13863	10.5 cm ²	22	730
13875	10.5	26	860
13882	10.5	21	700
13884	24.4	64	900

(a) Only fibers with length greater than 1 mm were counted.

TABLE II.-UNIFORMITY OF SINGLE CARBON FIBER DEPOSITION
AS COUNTED IN MULTIPLE ADJOINING AREAS ON SELECTED
SAMPLERS FROM THE ROUND ROBIN COUNTING

Test number	Sampler number (a)	Area counted	Fibers counted (b)	Projected total fibers on 15 x 23 cm sticky sampler	
				Number	Fraction of mean
BT-230	20013-1	40.3 mm ²	36	31100	1.044
	-2	↓	33	28500	0.956
BT-237	20812-1	↓	23	19900	1.181
	-2	↓	19	16400	0.973
	-3	↓	20	17300	1.027
	-4	↓	16	13800	0.819
BT-171	13884-R	12.2 cm ²	16	450	0.497
	-L	12.2 cm ²	48	1360	1.503

(a) Hyphenated suffix refers to adjoining areas shown in figure 2.

(b) Only fibers with length greater than 1 mm were counted.

TABLE III.-DISTRIBUTION OF SINGLE CARBON FIBERS BY LENGTH CATEGORY,
MEAN LENGTH, AND STANDARD DEVIATION BASED ON COUNTS OF FIBER
DEPOSITION ON STICKY SAMPLERS SELECTED FOR ROUND ROBIN
COUNTING FROM THREE NSWCBURN AND EXPLODE TESTS

Test & sampler number	Percentage of fibers of length category						Mean length mm	Std. dev. of length
	1- 1.9 mm	2- 2.9 mm	3- 3.9 mm	4- 4.9 mm	5- 5.9 mm	6- 6.9 mm		
BT-230 Heavy deposition								
19968	47	42	11	-	-	-	2.12	0.70
19970	34	44	11	9	2	-	2.41	.98
19980	73	19	5	3	-	-	1.77	.73
20013	66	25	9	-	-	-	1.80	.64
Average	55	33	9	2	1	-	2.02	
B-237 Medium deposition								
20803	72	19	3	3	3	-	1.79	0.94
20805	83	13	4	-	-	-	1.55	.57
20810	52	22	26	-	-	-	2.14	.97
20812	76	14	9	1	-	-	1.73	.76
Average	72	16	10	1	1	-	1.78	
BT-171 Light deposition								
13863	82	18	-	-	-	-	1.50	0.46
13875	92	4	-	-	-	4	1.56	1.02
13882	90	5	-	-	5	-	1.53	0.89
13884	81	14	3	-	2	-	1.54	0.74
Average	84	11	2	-	2	1	1.53	

TABLE IV.-ROUND ROBIN FIBER COUNTING OF TWELVE NSWC
CHAMBER BURN & EXPLODE TEST STICKY SAMPLERS REPORTED
AT LANGLEY RESEARCH CENTER MAY 10, 1979

Test & sampler number	Total single fibers on 15 x 23 cm sticky sampler as determined by participants*			
	DPG	LRC	SSI	TRW
BT-230 Heavy deposition				
19968	120885	45800	-	48869
19970	130959	40600	-	52016
19980	83320	32000	-	33794
20013	96254	29800	-	31475
Average	107854	37000		41538
BT-237 Medium deposition				
20803	74202	27600	40000	18341
20805	48464	19900	29100	15108
20810	44667	19900	23850	13358
20812	38610	16800	29800	17102
Average	51486	21000	30688	15977
BT-171 Light deposition				
13863	907	730	1088	622
13875	510	860	0	674
13882	477	700	816	499
13884	317	900	1474	407
Average	553	800	844	550

*Participating laboratories:

DPG US Army Dugway Proving Ground
LRC NASA Langley Research Center
SSI Scientific Service, Inc.
TRW Defense and Space Systems Group of TRW, Inc.

8583

Figure 1. - Photograph of a typical carbon fiber deposition from a burn and explode test in the NSWC chamber. Sticky sampler size 15 by 23 cm.

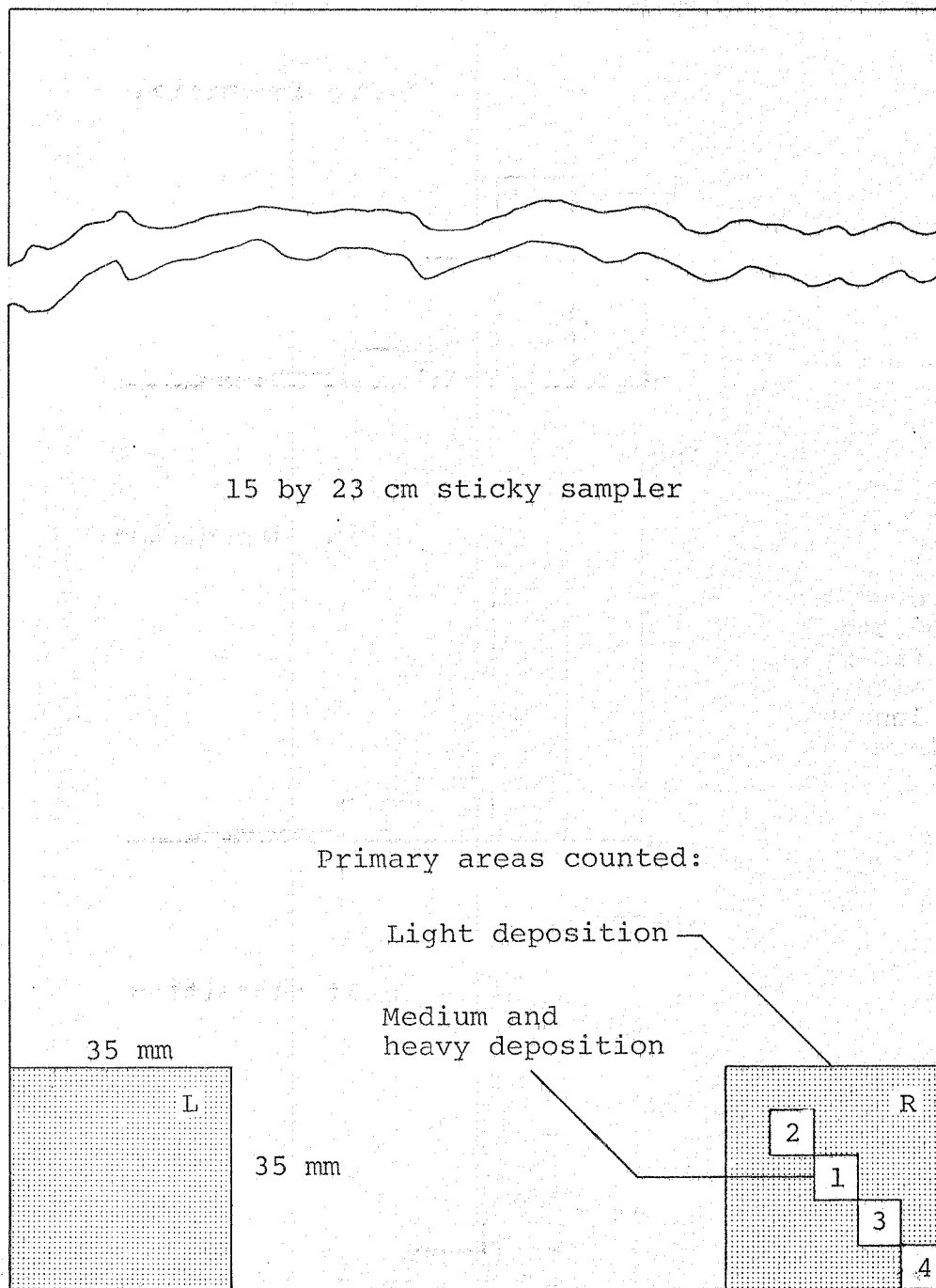


Figure 2. - Representative areas of sticky sampler that were counted for fiber deposition determination and for deposition uniformity comparisons on selected samplers.

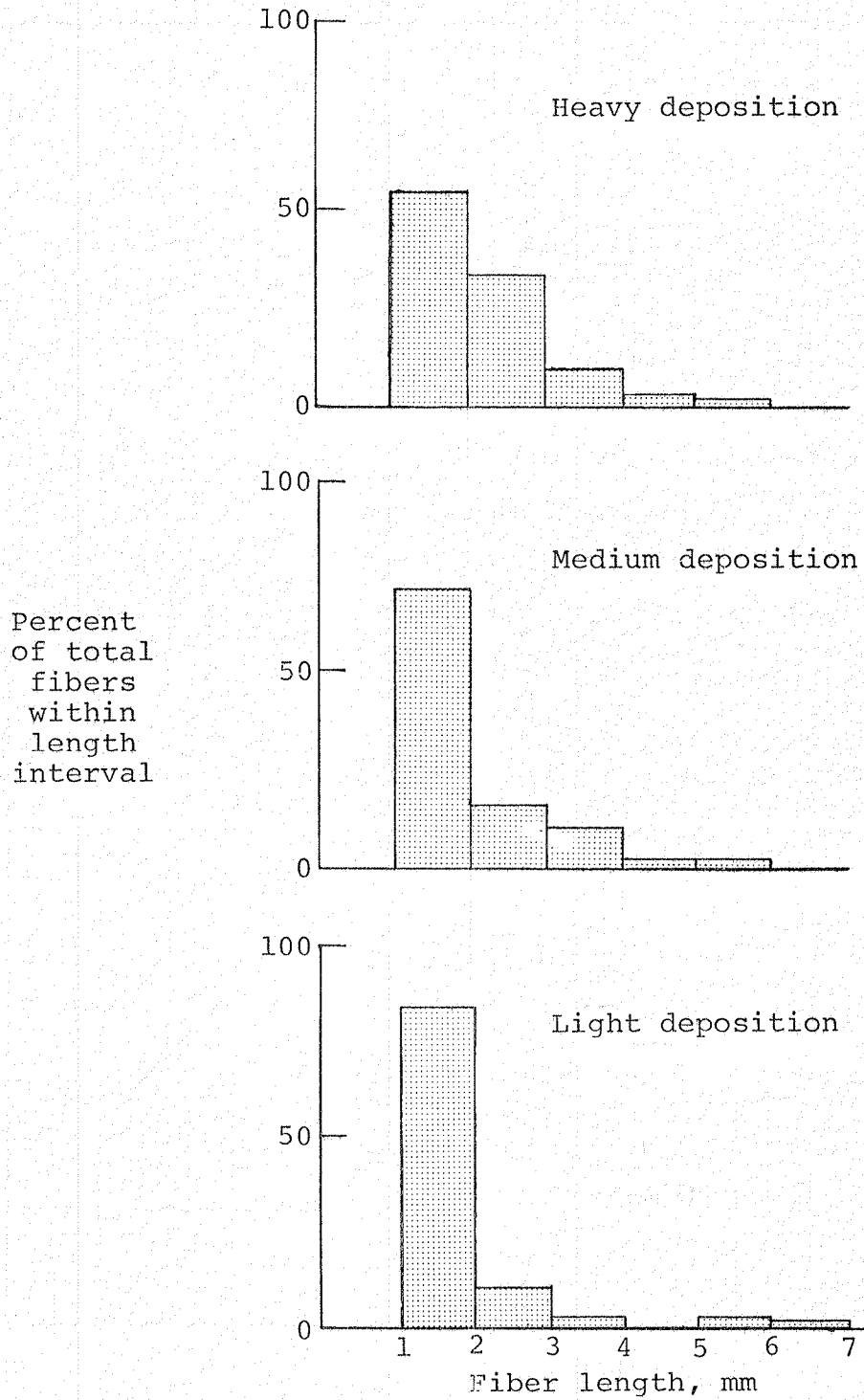


Figure 3. - Distribution of single carbon fibers by length for fiber lengths greater than 1 mm. Results from fiber deposits on sticky samplers selected for round robin counting.

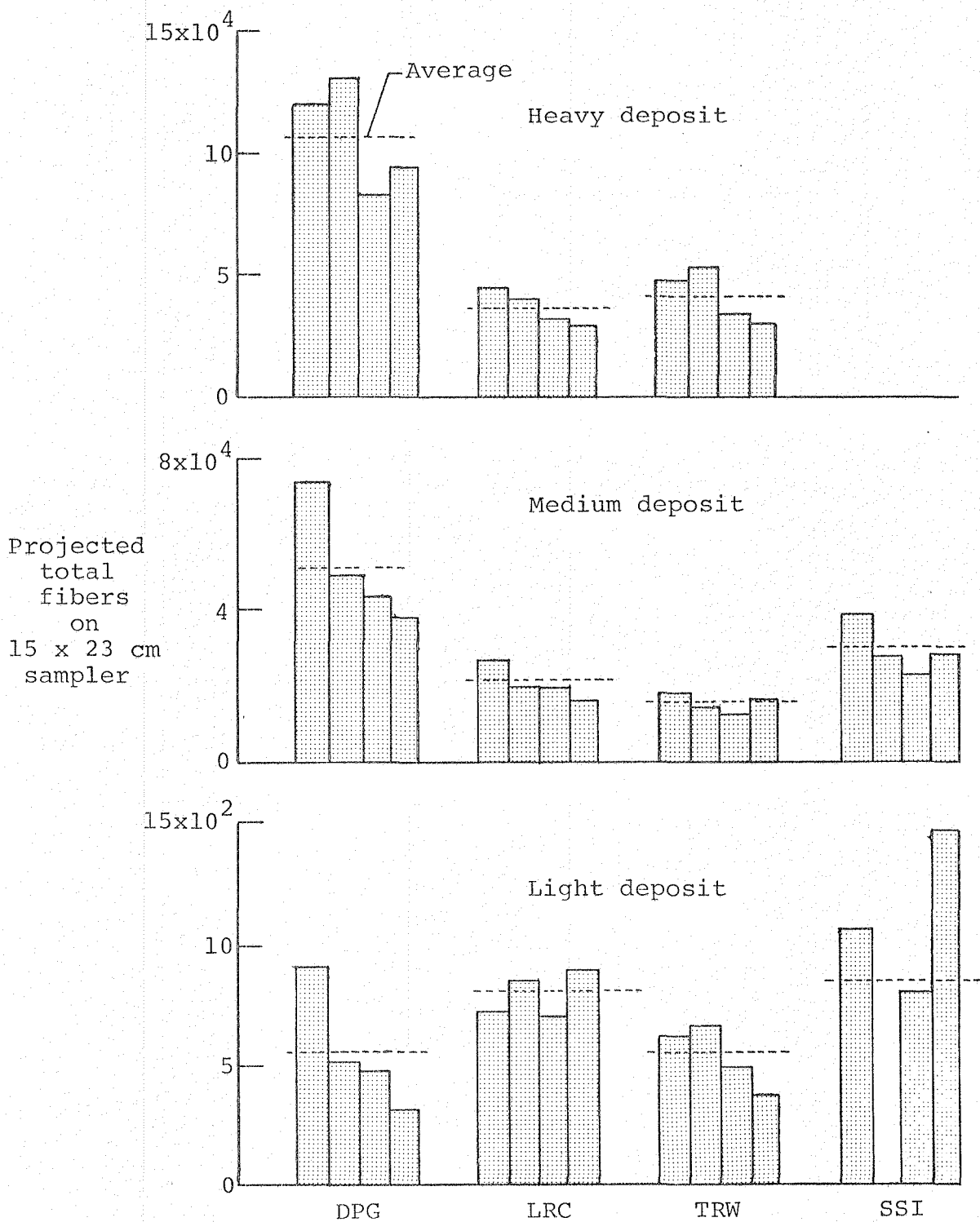


Figure 4. - Round robin fiber counting results from four laboratories counting from four samplers of each of three NSWC tests.

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